

Rapid solubilization of the treated pulp prior to spinning lyocell fibers is important in order to reduce the time required to generate lyocell fibers, or other molded bodies such as films, and hence reduce the cost of the process. Further, efficient dissolution is important because it minimizes the concentration of residual, undissolved particles, and partially dissolved, gelatinous material, which can reduce the speed at which fibers can be spun, tend to clog the spinnerets through which lyocell fibers are spun, and may cause breakage of the fibers as they are spun.

While not wishing to be bound by theory, it is believed that the processes of the present invention utilized to reduce the average D.P. of the cellulose also permeabilize the secondary layer of the pulp fibers, thereby permitting the efficient penetration of solvent throughout the pulp fiber. The secondary layer is the predominant layer of the cell wall and contains the most cellulose and hemicellulose.

Further, compositions of the present invention preferably have a carbonyl content of less than about 60 $\mu\text{mol/g}$ and a carboxyl content of less than about 60 $\mu\text{mol/g}$, more preferably, a carbonyl content of less than about 30 $\mu\text{mol/g}$ and a carboxyl content of less than 30 $\mu\text{mol/g}$. The carboxyl and carbonyl group content are measured by means of proprietary assays performed by Thuringisches Institut für Textil-und Kunststoff Forschung. V., Breitscheidstr. 97, D-07407 Rudolstadt, Germany. As an alternative to determining the carbonyl content of the pulp using the proprietary TITK assays, pulp samples and a thermal stable, low-carbonyl group pulp can be analyzed FTIR and the differences in the spectrums between the two samples can provide an indication of the existence of carbonyl groups.

Additionally, the treated pulp of the present invention preferably has a low transition metal content. Transition metals are undesirable in treated pulp because, for example, they accelerate the degradation of cellulose and NMMO in the lyocell process. Examples of transition metals commonly found in treated pulp derived from trees include iron, copper, nickel and manganese. Preferably, the total transition metal content of the compositions of the present invention is less than about 20 ppm, more preferably less than about 5 ppm. Preferably the iron content of the compositions of the present invention is less than about 4 ppm, more preferably less than about 2 ppm, as measured by Weyerhaeuser Test AM5-PULP-1/6010, and the copper content of the compositions of the present invention is preferably less than about 1.0 ppm, more

preferably less than about 0.5 ppm, as measured by Weyerhaeuser Test AM5-PULP-1/6010.

In order to make lyocell fibers, or other molded bodies, such as films, from the treated pulp of the present invention, the treated pulp is first dissolved in an amine oxide, preferably a tertiary amine oxide. Representative examples of amine oxide solvents useful in the practice of the present invention are set forth in U.S. Patent No. 5,409,532. The presently preferred amine oxide solvent is N—methyl-morpholine-N-oxide (NMMO). Other representative examples of solvents useful in the practice of the present invention include dimethylsulfoxide (D.M.S.O.), dimethylacetamide (D.M.A.C.), dimethylformamide (D.M.F.) and caprolactan derivatives. The treated pulp is dissolved in amine oxide solvent by any art-recognized means such as are set forth in U.S. Patent Nos. 5,534,113; 5,330,567 and 4,246,221. The dissolved, treated pulp is called dope. The dope is used to manufacture lyocell fibers, or other molded bodies, such as films, by a variety of techniques, including melt blowing, spun-bonding, centrifugal spinning, dry-jet, wet, and other methods. Examples of techniques for making a film from the compositions of the present invention are set forth in U.S. Patent No. 5,401,447 to Matsui et al., and in U.S. Patent No. 5,277,857 to Nicholson.

One useful technique for making lyocell fibers from dope involves extruding the dope through a die to form a plurality of filaments, washing the filaments to remove the solvent, and drying the lyocell filaments. FIGURE 2 shows a block diagram of the presently preferred process for forming lyocell fibers from the treated pulps of the present invention. The term "cellulose" in FIGURE 2 refers to the compositions of the present invention. If necessary, the cellulose in the form of treated pulp is physically broken down, for example by a shredder, before being dissolved in an amine oxide-water mixture to form a dope. The treated pulp of the present invention can be dissolved in an amine solvent by any known manner, *e.g.*, as taught in McCorsley U.S. Patent No. 4,246,221. The treated pulp can be wet in a nonsolvent mixture of about 40% NMMO and 60% water. The mixture can be mixed in a double arm sigma blade mixer and sufficient water distilled off to leave about 12-14% based on NMMO so that a cellulose solution is formed. Alternatively, NMMO of appropriate water content may be used initially to obviate the need for the vacuum distillation. This is a convenient way to prepare spinning dopes in the laboratory where commercially available NMMO

of about 40-60% concentration can be mixed with laboratory reagent NMMO having only about 3% water to produce a cellulose solvent having 7—15% water. Moisture normally present in the pulp should be accounted for in adjusting necessary water present in the solvent. Reference might be made to articles by Chanzy, H. and
5 A. Peguy, *Journal of Polymer Science, Polymer Physics Ed.* **18**:1137-1144 (1980), and Navard, P. and J.M. Haudin, *British Polymer Journal*, p. 174 (Dec. 1980) for laboratory preparation of cellulose dopes in NMMO water solvents.

The dissolved, treated pulp (now called the dope) is forced through extrusion orifices to produce latent filaments or fibers that are later regenerated.

10 FIGURE 3 and FIGURE 4 are scanning electron micrographs of a dry-jet, wet lyocell fiber of the present invention at 100X and 10,000X magnification respectively. The fibers shown in FIGURE 3 and FIGURE 4 were produced in accordance with Example 11.

Owing to the compositions from which they are produced, lyocell fibers
15 produced in accordance with the present invention have a hemicellulose content that is equal to or less than the hemicellulose content of the treated pulp that was used to make the lyocell fibers. Typically the lyocell fibers produced in accordance with the present invention have a hemicellulose content that is from about 0% to about 30.0% less than the hemicellulose content of the treated pulp that was used to make the lyocell fibers.
20 Lyocell fibers produced in accordance with the present invention have an average D.P. that is equal to, larger than or less than the average D.P. of the treated pulp that was used to make the lyocell fibers. Depending on the method that is used to form lyocell fibers, the average D.P. of the pulp may be further reduced during fiber formation, for example through the action of heat. Preferably the lyocell fibers produced in
25 accordance with the present invention have an average D.P. that is equal to, or from about 0% to about 20% less than or greater than, the average D.P. of the treated pulp that was used to make the lyocell fibers.

The lyocell fibers of the present invention exhibit numerous desirable properties. For example, lyocell fibers prepared from treated pulps of the present
30 invention comprise at least about 5 weight percent hemicellulose, cellulose having an average degree of polymerization from about 200 to about 1100, a copper number less than about 2.0 and a ΔR less than about 2.8. Preferably, such fibers have a